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GB 0813819 A JP 580171506 A
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(54) Abstract Title: **Making activated nickel and producing nickel carbonyl therefrom**

(57) A process for producing $\text{Ni}(\text{CO})_4$ from carbon monoxide and a source of nickel selected from elemental nickel, a nickel compound or mixtures thereof, provided the nickel compound is not nickel chloride per se or in admixture with a nickel carbonate ore, in an amount greater than 50% W/W nickel chloride; which process comprises (a) treating the nickel source with hydrogen at a pressure of at least atmospheric pressure and an effective temperature, in the presence of chloride anion or an in situ generator thereof precursor, to produce a resultant active nickel; (b) reacting the carbon monoxide with the resultant active nickel to produce the $\text{Ni}(\text{CO})_4$; and collecting the $\text{Ni}(\text{CO})_4$. The process offers the production of $\text{Ni}(\text{CO})_4$ at atmospheric pressure and at a sufficiently high rate for direct use in subsequent deposition processes without the need for storage facilities. Apparatus for the decomposition of the $\text{Ni}(\text{CO})_4$ so produced is also disclosed.

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This print takes account of replacement documents submitted after the date of filing to enable the application to comply with the formal requirements of the Patents Rules 1995

Original Printed on Recycled Paper

Figure 1

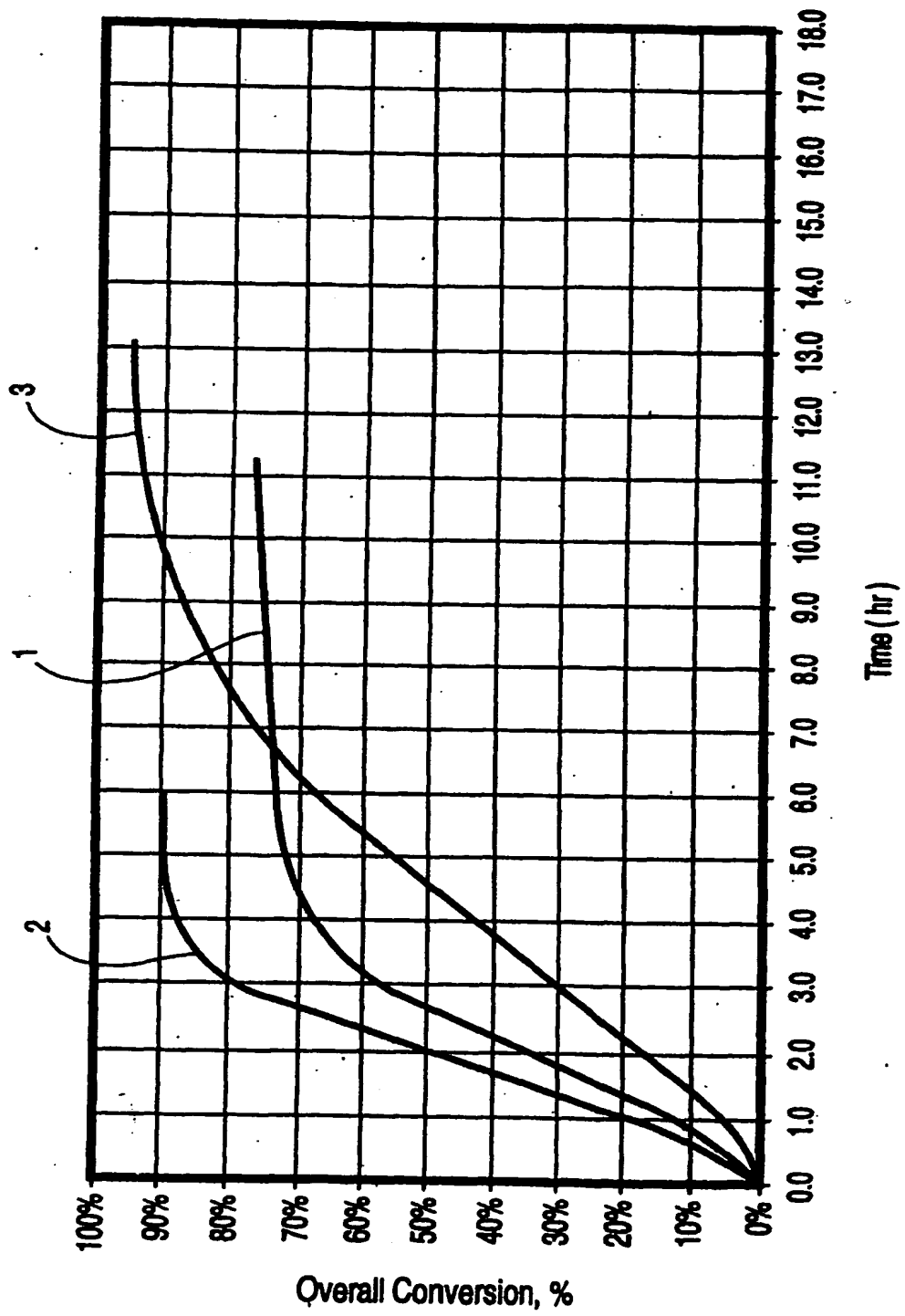


Figure 2

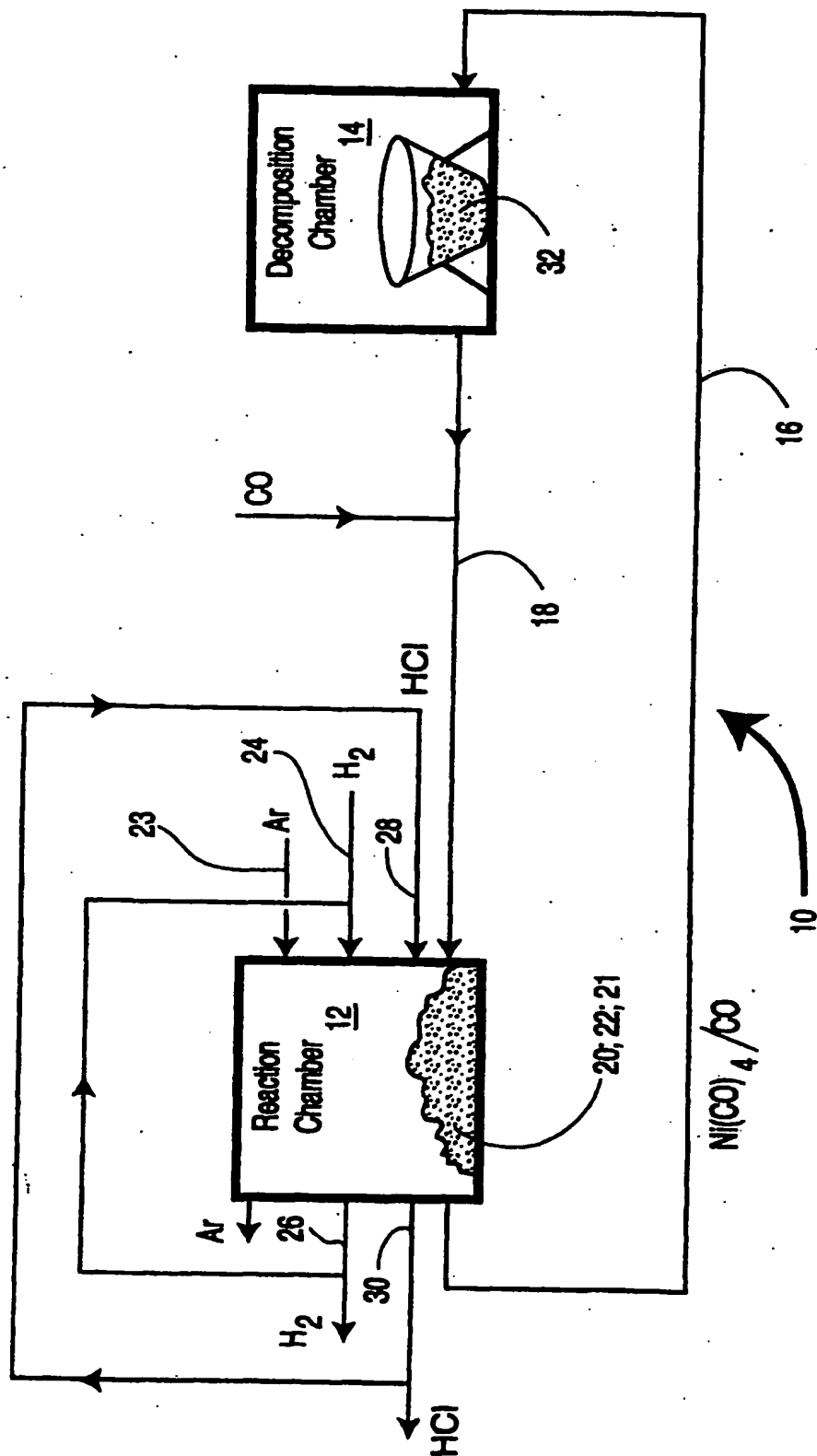
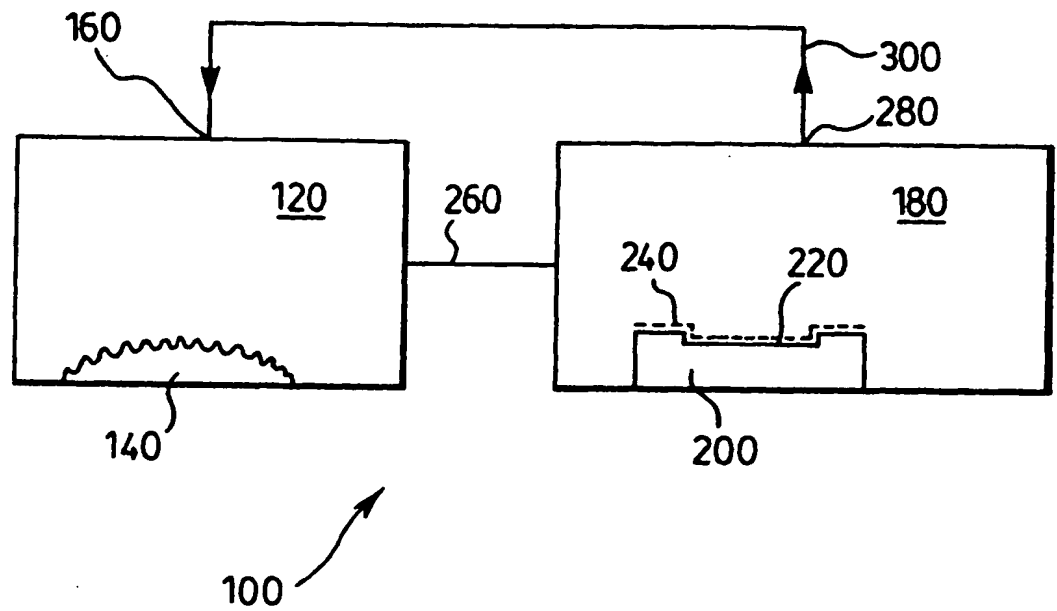


FIG. 3.



PROCESS FOR PRODUCING NICKEL CARBONYL,
NICKEL POWDER AND USE THEREOF

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FIELD OF THE INVENTION

This invention relates to processes for producing nickel carbonyl, more particularly to producing nickel powders of use in producing said nickel carbonyl by reaction with carbon monoxide, and to said nickel powders made by said process.

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BACKGROUND TO THE INVENTION

Nickel carbonyl, $\text{Ni}(\text{CO})_4$, was first produced by the reaction of metallic nickel with carbon monoxide by Mond in the early part of the 19th century. Today, one of the major industrial processes for making metallic nickel is based on the production of $\text{Ni}(\text{CO})_4$ and subsequent thermal decomposition thereof to Ni and CO. One known commercial process operates at about 180°C and a CO pressure of about 70 atm. It is known that the CO pressure may be reduced when the reactant nickel is catalytically activated.

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Activation of the metal has been observed in the presence of mercury (1, 2), sulfur in the form of H_2S (3, 4), hydrogen (5, 6) or carbon (7). It has been suggested that the high initial rate of formation of $\text{Ni}(\text{CO})_4$ and the subsequent decline to a steady state value is the result of a rapid decrease in the number of activated reaction sites which are produced upon heat treatment of the sample (8, 9, 6). A study of surface changes during carbonyl synthesis suggests that the maximum rate is associated with fundamental changes in the defect structure. All of the above methods use catalytic activation of nickel in the presence of CO.

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However, it can be readily appreciated that processes that at atmospheric pressure can produce nickel, particularly, activated nickel for subsequent reaction with CO at atmospheric pressure would provide significant capital and operating cost advantages.

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Further, it can also be appreciated that processes that enable $\text{Ni}(\text{CO})_4$ to be manufactured at a sufficient rate as to obviate the need for storage in order to build up

a sufficient supply for practical, efficient use in a subsequent nickel deposition process, would also offer significant capital and operating cost savings. To-date, in commercial operations rate limitations on the production of $\text{Ni}(\text{CO})_4$ require such storage facilities and operations.

5 There is, thus, a desire for an improved method of $\text{Ni}(\text{CO})_4$ production which is operable at atmospheric pressure and which is of a sufficient rate as to negate the need for storage of the $\text{Ni}(\text{CO})_4$ prior to use in a subsequent decomposition and/or deposition process.

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SUMMARY OF THE INVENTION

It is an object of the present invention to provide a process for the commercial production of $\text{Ni}(\text{CO})_4$ from a source of nickel in an efficacious manner at atmospheric pressure, with resultant capital and operating cost savings.

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It is a further object of the present invention to provide a process and apparatus for the commercial continuous production of $\text{Ni}(\text{CO})_4$ from a source of nickel at a sufficiently high rate as to negate the need for storage of the $\text{Ni}(\text{CO})_4$ prior to a subsequent decomposition step, with resultant capital and operating cost savings.

Accordingly, in one aspect the invention provides a process for producing $\text{Ni}(\text{CO})_4$ from carbon monoxide and a source of nickel selected from the group consisting of elemental nickel, a nickel compound or mixtures thereof, provided said nickel compound is not nickel chloride per se or in admixture with a nickel carbonate ore in an amount greater than 50% w/w nickel chloride which process comprises (a) treating said nickel source with hydrogen at a pressure of at least atmospheric pressure and an effective temperature, in the presence of chloride anion or an in situ generator thereof precursor; to produce a resultant active nickel; and (b) reacting said carbon monoxide with said resultant active nickel to produce said $\text{Ni}(\text{CO})_4$.

By the term "resultant active nickel" as used in this specification and claims is meant resultant particulate nickel that reacts with CO at essentially atmospheric pressure and a temperature of from ambient (20°C) to 80°C to effect conversion to $\text{Ni}(\text{CO})_4$ at an acceptable conversion rate.

By the term "acceptable conversion rate" is meant herein the rate of production of $\text{Ni}(\text{CO})_4$ in the order of at least 0.1 g/hr $\text{Ni}(\text{CO})_4$ per g/Ni, which provides an efficacious rate for direct utilization, without the need for a build-up in storage, preferably for use in a commercial subsequent deposition process. A more acceptable rate is 1 g/hr $\text{Ni}(\text{CO})_4$ per g/Ni and a still more advantageous rate is the maximum rate of 3.0 g/hr. $\text{Ni}(\text{CO})_4$ per g/Ni.

The effective temperature is a temperature which effects the production of resultant active nickel at an acceptable rate of at least atmospheric pressure. Preferably, the effective temperature is in the range $300^\circ - 650^\circ\text{C}$ and more preferably, $350^\circ - 550^\circ\text{C}$.

The $\text{Ni}(\text{CO})_4$ produced in step (b) may be collected, or, alternatively, when made at an acceptable conversion rate, as herein defined, preferably of at least 0.25 g/hr $\text{Ni}(\text{CO})_4$ per g Ni, preferably, at a rate of 3.0 g/hr $\text{Ni}(\text{CO})_4$ per g Ni directly passed to a deposition chamber for immediate decomposition to Ni and CO. This enables the CO to be immediately recycled in a closed-loop manner as to provide a continuous CO closed-loop process.

Accordingly, in a further aspect, the invention provides an improved apparatus for the production of nickel powder, coatings, articles or compounds from the decomposition of $\text{Ni}(\text{CO})_4$, said apparatus comprising:-

- (a) a decomposition chamber having a $\text{Ni}(\text{CO})_4$ feed inlet, a spent CO outlet and adapted to receive a substrate;
- (b) a $\text{Ni}(\text{CO})_4$ production chamber having a CO feed inlet, a gaseous $\text{Ni}(\text{CO})_4$ product outlet and adapted to receive source nickel for reaction with CO, wherein the improved apparatus comprises
- (i) $\text{Ni}(\text{CO})_4$ direct feed means between said $\text{Ni}(\text{CO})_4$ feed inlet of said decomposition chamber for feeding $\text{Ni}(\text{CO})_4$ directly from said production chamber to said decomposition chamber at an acceptable feed rate;
- (ii) CO recycle conduit means between said spent CO outlet and said CO feed inlet; and
- (iii) wherein said source nickel comprises activated nickel produced as hereinabove defined.

Present prior art processes have very low conversion rates, typically of less than 0.03 g $\text{Ni}(\text{CO})_4$ /hr. per g/Ni, which requires that the $\text{Ni}(\text{CO})_4$ needs to be stored to a required volume prior to use in a subsequent decomposition or deposition step.

The nickel compounds of use in the practise of the invention as hereinabove defined may readily be selected, from, but not limited to, for example, the group consisting of a nickel salt, most particularly, nickel chloride, carbonate, hydroxide, oxide and metallic elemental nickel. The metallic elemental nickel is most preferably in particulate form, for example, as a very fine powder.

The preferred nickel salt is nickel chloride, preferably in the form of a hydrate, or a mixture thereof with nickel carbonate in the form of zaraitite ($2\text{Ni}(\text{OH})_2 \cdot \text{NiCO}_3 \cdot 4\text{H}_2\text{O}$), preferably wherein the amount of nickel chloride is such as to produce 20 - 25% W/W of chloride based on nickel in the mixture.

The chloride anion may be selected from, by way of example, but not limited to, hydrogen chloride and a metallic chloride salt, such as, for example, an alkali, alkaline earth or transition metal simple or complex salt, e.g. FeCl_3 . The invention also includes processes that involve the use of precursors of chloride ion under the reaction conditions defined, such as, for example, suitable use of Cl_2 , oxides of chlorine gas and OCl_2 salts that produce chloride anion in situ.

The chloride anion is, preferably, present at a ratio of at least 1:10 atomic W/W% Cl to Ni, more preferably 1:5 atomic W/W%.

A preferred process is wherein the chloride anion is present as gaseous hydrochloric acid in gaseous admixture with the hydrogen, and more preferably, wherein the nickel compound is first treated with hydrogen at the effective temperature for a first period of time and subsequently treated with the gaseous admixture for a second period of time, at the effective temperature.

The chloride anion in alternative embodiments may be generated in situ under the aforesaid process conditions, according to the invention as defined, in requisite effective amounts from chloride anion generating precursors, such as, for example, chlorate compounds and chlorine gas.

In a further aspect, the invention provides the resultant active nickel when made by a process as hereinabove defined prior to its subsequent reaction with CO to form $\text{Ni}(\text{CO})_4$.

In a yet further aspect, the invention provides a process as hereinabove defined for producing resultant nickel from the decomposition of nickel carbonyl produced by a process as hereinabove defined.

We have found, further, that relatively small amounts of metal chlorides, e.g. ferric chloride in the presence of non-chloride nickel compounds enable activated nickel to be formed according to the process of the invention as hereinabove defined.

The gaseous product stream comprises H_2 and HCl and, optionally, H_2O , CO_2 and CO.

BRIEF DESCRIPTION OF THE DRAWINGS

In order that the invention may be better understood, preferred embodiments will now be described by way of example only with reference to the accompanying drawing, wherein:-

Fig. 1 is a graph showing overall conversion (%) against reaction time (hr.) for various processes according to the invention;

Fig. 2 is a diagrammatic flow diagram of a continuous self-contained process according to the invention; and

Fig. 3 is a diagrammatic sketch of a $\text{Ni}(\text{CO})_4$ production reaction chamber in direct communication with a Ni deposition chamber according to the invention.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

In order that the invention may be better understood, preferred embodiments will now be described by way of example only, wherein Examples 1 and 2 do not constitute part of the invention as claimed but are provided for comparison purposes only.

Example 1 (Prior Art)

NiCO₃ powder (200 g) was placed in an extraction reactor and treated with a stream of H₂ gas at 300 mL/min flow rate, at 500°C for 7 hours to effect substantially complete reduction. The nickel powder produced was cooled to 100°C and the atmosphere of H₂ was subsequently replaced with carbon monoxide. The reactor was cooled further to 50°C and CO gas passed through at 300 mL/min flow rate. The resulting Ni(CO)₄ was passed through a pair of carbonyl decomposers according to the prior art and nickel was recovered as nickel powder, (10 g; 10 % yield) after 12 hours.

Example 2 (Prior Art)

Ni(OH)₂ powder (100 g) was placed in the extraction reactor and treated with a stream of H₂ gas at 300 mL/min flow, at 500°C for 7 hours to essentially complete reduction. The resulting nickel powder was cooled to 100°C in the atmosphere of H₂ which was subsequently replaced with carbon monoxide. The reactor was cooled down further to 50°C and CO gas passed through at 300 mL/min flow rate. The resulting Ni(CO)₄ gas was passed through carbonyl decomposers and nickel powder (6g, 9.5 yield) after 12 hours was recovered.

Example 3

300.1g of a nickel carbonate/ nickel chloride mixture (10:1 w/w) was placed into an extraction reactor and treated with hydrogen (2L/min) at 450°C for 6 hours. Subsequently, the hydrogen was replaced with argon, the reactor cooled to 40°C and the argon replaced with carbon monoxide at a gas temperature of 80°C, and flow rate of 4 L/min. whereby nickel carbonyl was formed, collected and subsequently decomposed to Ni and CO to provide (103g; 70%) yield of nickel extraction yield in 6 hr.

Example 4

302.3 g of the same mixture as in Example 3 was treated under similar conditions but wherein after 0.5 h, the hydrogen gas was doped with 1% of HCl for a further reaction period of 4 hours. The subsequent nickel extraction procedure was similar to Example 3 and gave 134g; 90% yield of nickel in 6 h.

Example 5

300.1 g of nickel carbonate was treated under similar conditions as in Example 4 but wherein after 0.5 h a flow of 900cc/min of HCl gas was introduced into the hydrogen flow at 2 L/min for 4 h. The subsequent nickel extraction procedure was similar to Example 3 and gave a 96.48 % yield of nickel in 13 h.

The aforesaid examples 3-5 are better illustrated with reference to Fig. 1 wherein:-

Line 1 represents the carbonylation of nickel produced by the reaction of a mixture of nickel carbonate/nickel chloride 10:1. (6 h, 70.4% yield) according to Example 3.

Line 2 represents the same composition according to line 1 plus 1% pf HCl in the gas stream (6 hours, 90.45% yield) according to Example 4; and

Line 3 represents 100% nickel carbonate plus HCl (13 h, 96.48%) according to Example 5.

The aforesaid examples clearly illustrate the beneficial effect of having chloride anion present in admixture with a nickel compound in the hydrogen reactor in producing a particulate nickel more efficacious in reacting with CO to produce $\text{Ni}(\text{CO})_4$.

TABLE 1

Example	Ni Compound (g)	Equivalent Ni(g)	Deposited Ni(g)	Time (hr)*	Rate** gNi/gNi/hr (approx.)	Rate*** $\text{Ni}(\text{CO})_4$ /gNi/hr
#1	200 (NiCO_3)	98	10	12	0.01	0.3
#2	100 ($\text{Ni}(\text{OH}_2)$)	63	6	12	0.01	0.03
#3	300 NiCO_3 / Ni Cl_2 (10:1)	147	103	6	0.12	0.36
#4	302 NiCO_3 / Ni Cl_2 (10:1)	148	134	6	0.15	0.45
#5	300 g NiCO_3	148	142	13	0.07	0.21

- * carbonylation and subsequent decomposition time from treated (reduced) Ni.
- ** rate of deposition of Ni metal per se per hour per 1 gm equivalent Ni, calculated from Ni compound starting material.

5 *** Rate of production of $\text{Ni}(\text{CO})_4$ per hr. per g Ni.

Table 1 shows the beneficial enhancement in the rate of production of Ni from its various sources by the process according to the present invention, wherein the presence of chloride anion in Examples 3, 4 and 5 shows the very significant beneficial effect over the absence of chloride anion in Examples 1 and 2.

10 This enhancement in production rate of $\text{Ni}(\text{CO})_4$ enables the direct use thereof in any subsequent desired decomposition step.

Fig. 2 is a diagrammatic flow diagram of a continuous nickel deposition process self-contained with respect to CO, according to the invention. It shows generally as 10, a reaction chamber 12 linked to decomposition chamber 14 by
15 $\text{Ni}(\text{CO})_4$ and CO conduits 16 and 18, respectively.

Chamber 12 contains, alternatively, nickel source 20 and resultant nickel 22; and has hydrogen feed and outlet/recycle conduits 24 and 26, respectively; HCl feed and outlet/recycle conduits 28 and 30, respectively; and $\text{Ni}(\text{CO})_4$ /CO exit conduit 16. Decomposition chamber 14 contains a substrate 32 to be nickel coated from line 16.

20 In operation, nickel source 20 is treated with hydrogen, typically, at 400-500 °C for 5-15 hours and 2 l/min at atmosphere pressure to produce a reduced nickel powder 21.

HCl gas at 1 l/min and 50 - 80° is then recycled through chamber 12, optionally, with hydrogen, to produce treated nickel powder 22. Chamber 12 is then
25 subsequently purged with, for example, argon from conduit 23 and, thereafter, CO from conduit 18 is fed into chamber 12, wherein $\text{Ni}(\text{CO})_4$ is produced and passed through conduit 16 to decomposition chamber 14. Recycle conduit as shown in Fig. 2 are utilized as desired.

It can be seen that once the process is operating at "steady state" for an
30 alternative two-stage operative cycle, that the amount of CO used in the production of $\text{Ni}(\text{CO})_4$ can be met from the decomposition thereof in chamber 14. The process can thus be considered as being essentially self-contained with respect to CO.

Importantly, since the rate of production of $\text{Ni}(\text{CO})_4$ in chamber 12 is sufficiently high enough to warrant a direct feed to chamber 14 for decomposition of Ni onto substrate 32 in an efficacious manner, no intervening storage facility is required. This is of value in commercial operations.

5 Fig. 3 shows generally as 100 a double chamber apparatus for the continuous production and decomposition of $\text{Ni}(\text{CO})_4$.

$\text{Ni}(\text{CO})_4$ production chamber 120 contains activated nickel 140 prepared according to the invention, either in an initial starting amount as shown in this embodiment or under continuous feed means of an alternative embodiment (not shown). Chamber 120 has a CO feed inlet 160, whereby in operation CO gas reacts
10 with nickel 140 to produce $\text{Ni}(\text{CO})_4$ at an acceptable production rate selected from 0.1 to 3.0 g $\text{Ni}(\text{CO})_4$ /g activated Ni/hr.

Decomposition chamber 180 contains a mandrel, mold or like substrate 200 having a substrate surface 220 upon which is deposited Ni as a coating 240 which
15 constitutes a Ni shell product in the embodiment shown by decomposition of gaseous $\text{Ni}(\text{CO})_4$ from chamber 120 fed through conduit 260. By-product CO exits chamber 180 through outlet 280 and is recycled through conduit 300 to feed inlet 160. The above process constitutes a continuous, CO self-contained system wherein the $\text{Ni}(\text{CO})_4$ is essentially produced in chamber 120 at a rate sufficient to provide for the
20 practical deposition operation in chamber 180 as to negate the need for $\text{Ni}(\text{CO})_4$ storage with its attendant capital and operating costs.

Although this disclosure has described and illustrated certain preferred embodiments of the invention, it is to be understood that the invention is not restricted to those particular embodiments. Rather, the invention includes all embodiments
25 which are functional or mechanical equivalents of the specific embodiments and features that have been described and illustrated.

Claims

1. A process for producing $\text{Ni}(\text{CO})_4$ from carbon monoxide and a source of nickel selected from the group consisting of elemental nickel, a nickel compound or mixtures thereof, provided said nickel compound is not nickel chloride per se or in admixture with a nickel carbonate ore in an amount greater than 50% w/w nickel chloride which process comprises (a) treating said nickel source with hydrogen at a pressure of at least atmospheric pressure and an effective temperature, in the presence of chloride anion or an in situ generator thereof precursor; to produce a resultant active nickel; and (b) reacting said carbon monoxide with said resultant active nickel to produce said $\text{Ni}(\text{CO})_4$.
2. A process as defined in claim 1 wherein said nickel compound is selected from the group consisting of a nickel salt, nickel hydroxide, nickel carbonate and nickel oxide.
3. A process as defined in claim 1 or claim 2 wherein said chloride anion is present from a compound selected from hydrogen chloride and a metallic chloride.
4. A process as defined in any one of claims 1 to 3 wherein said metallic chloride is selected from the group consisting of an alkali, alkaline earth and transition metal chloride.
5. A process as defined in any one of claims 1 to 4 wherein said chloride anion is present as gaseous hydrochloric acid in gaseous admixture with said hydrogen.
6. A process as defined in any one of claims 1 to 5 wherein said nickel compound is first treated with hydrogen at said effective temperature for a first period of time and subsequently treated with said gaseous admixture for a second period of time, at said effective temperature.
7. A process as defined in claim 5 or claim 6 wherein said nickel compound comprises nickel carbonate and said gaseous admixture comprises HCl and H_2 in the molar ratio of about 1:2.
8. A process as defined in any one of claims 1 to 7 wherein said nickel compound is nickel carbonate.
9. A process as defined in any one of claims 1 to 8 wherein said nickel compound is nickel chloride.

10. A process as defined in any one of claims 1 to 9 wherein said effective temperature is selected from the range 300° - 650°C.
11. A process as defined in claim 10 wherein said effective temperature is selected from 350° - 550°C.
- 5 12. A process as defined in any one of claims 1 to 11 wherein said resultant active nickel is reacted with carbon monoxide at a temperature of about 50°C.
13. A process as defined in any one of claims 1 to 12 wherein said $\text{Ni}(\text{CO})_4$ is produced in step (b) at an acceptable conversion rate, and further comprising passing said $\text{Ni}(\text{CO})_4$ directly to a decomposition chamber and decomposing
- 10 said $\text{Ni}(\text{CO})_4$ to deposit nickel and CO.
14. A process as defined in claim 13, wherein said decomposition effects deposition of Ni.
15. A process as defined in claim 13 or claim 14 wherein said acceptable conversion rate produce $\text{Ni}(\text{CO})_4$ at a rate of at least 0.1 g/hr $\text{Ni}(\text{CO})_4$ per g Ni.
- 15 16. A process as defined in claim 15 wherein said acceptable conversion rate is at least 0.25 g/hr $\text{Ni}(\text{CO})_4$ per g Ni.
17. A process as defined in claim 16 wherein said acceptable conversion rate produces $\text{Ni}(\text{CO})_4$ at a rate of 3.0 g/hr. $\text{Ni}(\text{CO})_4$ per g Ni.
18. A process as defined in any one of claims 1 to 17 wherein said precursor is selected from the group consisting of Cl_2 , oxides of chlorine and NaOCl .
- 20 19. A process for producing a resultant active nickel compound of subsequent use in the production of $\text{Ni}(\text{CO})_4$ by reaction with carbon monoxide, from a nickel source selected from the group consisting of elemental nickel, a nickel compound or mixtures thereof, which process comprises (a) treating said nickel source with hydrogen at a pressure of at least atmospheric pressure and an effective temperature, in the presence of chloride anion or an in situ generator thereof precursor as defined in any one of claims 1 to 18; and collecting said resultant active nickel.
- 25 20. A resultant nickel produced by a process as defined in any one of claims 13 to 17.
- 30 21. An improved apparatus for the production of nickel powder, coatings, articles or compounds from the decomposition of $\text{Ni}(\text{CO})_4$, said apparatus comprising:-

- (a) a decomposition chamber having a $\text{Ni}(\text{CO})_4$ feed inlet, a spent CO outlet and adapted to receive a substrate;
- (b) a $\text{Ni}(\text{CO})_4$ production chamber having a CO feed inlet, a gaseous $\text{Ni}(\text{CO})_4$ product outlet and adapted to receive source nickel for reaction with CO, wherein the improved apparatus comprises
- (i) $\text{Ni}(\text{CO})_4$ direct feed means between said $\text{Ni}(\text{CO})_4$ feed inlet of said decomposition chamber for feeding $\text{Ni}(\text{CO})_4$ directly from said production chamber to said decomposition chamber at an acceptable feed rate;
- (ii) CO recycle conduit means between said spent CO outlet and said CO feed inlet; and
- (iii) wherein said source nickel comprises activated nickel produced according to any one of claims 1 to 20.
22. Apparatus as defined in claim 21 wherein said direct feed means comprises means for feeding said $\text{Ni}(\text{CO})_4$ to said decomposition chamber at a rate of at least 0.1 g/hr. $\text{Ni}(\text{CO})_4$ per g said source nickel.



INVESTOR IN PEOPLE

Application No: GB 0406594.2
Claims searched: 1-22

Examiner: Colin Clarke
Date of search: 10 June 2004

Patents Act 1977 : Search Report under Section 17

Documents considered to be relevant:

Category	Relevant to claims	Identity of document and passage or figure of particular relevance	
X	1,9 & 19 at least	GB 0813819	MONTECATINI see whole document
X	19	GB 1401718	INTERNATIONAL NICKEL CO OF CANADA see p1 lines 68-86
X	19	GB 1099464	RESEARCH COUNCIL OF ALBERTA see claim 1 & example 3
X	19	JP 58-171506 A	SUMITOMO METAL MINING see Derwent Abstract 1983-817737
X	19	Derwent Abstract 1981-10940D & JP56-2136B SUWA SEIKOSHA	

Categories:

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

Field of Search:

Search of GB, EP, WO & US patent documents classified in the following areas of the UKCTM:

C1A

Worldwide search of patent documents classified in the following areas of the IPC⁷:

C01G;C22B

The following online and other databases have been used in the preparation of this search report:

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